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Properties of slurries made of fast pyrolysis oil and char or beech wood

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ABSTRACT

The properties of slurries made of pyrolysis oil mixed with wood, char or ground char were investigated with respect to phase transitions, rheological properties, elemental compositions, and energy density. Also the pumping properties of the slurries were investigated at temperatures of 25, 40 and 60 °C and solid loadings from 0 to 20 wt%. The phase transitions of the wood slurry samples were observed at lower solid loadings compared to the char slurry samples. The apparent viscosity of the slurry samples was found to be considerably impacted by solid loading (0–20 wt%) and temperature (25–60 °C), especially in the phase transition region. The slurry viscosities with 20 wt% char loading, 20 wt% ground char loading and 15 wt% wood loading (at a shear rate of 100 s^{-1}) are 0.7, 1.0 and 1.7 Pa.s, respectively at 60 °C and these values increases 1.2–1.4 times at 40 °C and 3–4 times at 25 °C. The wood, char and ground char slurry samples with 5–20 wt% solid loading obtain a volumetric energy density of 21–23 GJ/m^3. The slurry sample with 20 wt% ground char having a d_{80} of 118 μm was pumped successfully into a pressurized chamber (0–6 bar) while plugging appeared when the slurry samples with 15 wt% char having a d_{80} of 276 μm was pumped into the pressurized chamber.

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1. Introduction

Using biomass as a feedstock for boilers and gasifiers is a considerable challenge since biomass has a low bulk density and a low energy density [1,2] that causes high transportation cost [3] and high processing cost. Besides, the energy consumption needed to grind the biomass to a suitable size for suspension boilers or entrained flow gasifiers is relatively high [4,5].

Entrained flow pressurized gasification has the capability for converting biomass to a tar free syngas with a high concentration of H_2 and CO (50% CO and 30% H_2) [6,7], and a high fuel carbon conversion (>99%) can be obtained. However, using a high amount of auxiliary gases in the hopper system when feeding solid fuels into a pressurized gasifier cause a reduced efficiency, high complexities, and high cost [4,7,8]. In contrast pumping of viscous slurries such as fly ash-water mixtures [9], or bio-oil-char mixtures (bioslurry) [5,6] into a high pressure reactor is known to be a simple, reliable and do not cause a large use of auxiliary gases. The use of a slurry as feedstock for a pressurized gasifier probably reduce operating costs and operating problems [4] compared to that of raw solid biomass.

Fast pyrolysis of biomass often provides bio-oil yields of 55–75 wt%, char yields of 10–20 wt% and gas yields of 10–20% wt [10]. The produced gas that contains about 10% of the biomass energy content is often proposed to be used as heat

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source for the fast pyrolysis process. The char and bio-oil may contain approximately 90% of the biomass heating value [5]. A bioslurry with 20% char content has a density of around 1250 kg/m^3 and an energy density of about 23 GJ/m^3 [11]. This is 3 times the bulk density and 4 times the energy density of that of biomass [11]. Fast pyrolysis can be considered as a densification step to supply a slurry for gasification and thereby reduce the costs of gasifier operation, as well as reducing cost for transportation and milling of the biomass. Power consumption for milling of biomass to a final average sizes from 0.2 to 0.6 mm is higher in a range of from 3 to 5 times than that of milling pyrolytic char [4]. Recently considerable efforts to make a commercial bioslurry have gained attention by several organizations [5,10]. Dynamotive has built a fast pyrolysis 200 tpd plant in Guelph (2007) for producing bioslurry (BioOil Plus) [12] as fuel for boilers, gasifiers and kiln furnaces. Karlsruhe Institute of Technology (Germany) (KIT) has installed a two-MWth fast pyrolysis plant and a five-MWth gasifier (2011) [5], for which the slurry is used as a feedstock for gasification to produce gasoline.

Some information on bioslurry properties (mixture of bio-oil and char) is provided by Abdullah et al [11] and Dahmen et al. [5]. Abdullah et al [11]. characterized slurry properties (from mallee bio-oil and its pyrolytic char) with respect to rheological properties, elemental content, static stability, and volumetric energy density with char loading from 8 to 20 wt% at 25 and 50 °C. The results demonstrated that the slurry has a non-Newtonian characteristic with pseudo-plastic behavior that has a good pumpability. The slurry properties are considerably influenced by the char loadings and the temperature. A maximum slurry viscosity of 0.5 Pa.s for a slurry with 20 wt% char loading (having d_{80} of 74 μm) was obtained at a temperature of 25 °C.

Although bioslurry is a promising feedstock for pressurized gasification [5,6], an overall investigation of bioslurry properties as a pressurized gasification feedstock has not been performed. Furthermore, using same wood to produce the bioslurry is also an interesting concept that could ensure that a maximum amount of slurry is made based on the amount of bio-oil that is produced. The fine char is mixed with the bio-oil and if possible residual oil is mixed with wood. However, to date, no data on bioslurry with wood is mentioned in the literature. Thus the objectives of this work are to study the behavior of bioslurry as a feedstock for pressurized gasification with respect to visually observed phase transition, elemental compositions, energy densification, rheological properties, and possibilities to pump the slurry at pressurized conditions. Slurries made by mixing pyrolysis oil with beech wood, char (from the fast pyrolysis process) and ground char were investigated and the feasibility of the feedstocks was evaluated. Furthermore, the data also provide important properties of bioslurries that also can be used as a feedstock for boilers and atmospheric gasifiers.

2. Experimental section

2.1. Materials and method

Bio-oil and char were produced from pyrolysis of Danish beech wood on a pyrolysis centrifugal reactor (PCR) at 550 °C [13]. The yields of 69 wt% (as received) for bio-oil, 14 wt% (as received) for char and 16 wt% "as received" for gas were obtained at the applied temperature [13]. If all the char and bio-oil products were used to produce a bioslurry, the slurry would have a char concentration of 17 wt% and contain 88% energy contents of the beech wood (information on the energy recovery is presented in reference 13). The bio-oil has a water content of 25 wt% and a higher heating value (HHV) of 18.3 MJ/kg "as received". Ground char was produced by milling of the pyrolysis char. Bioslurry samples with the wood (moisture content of 9 wt%), the char (moisture content less than 0.5 wt%) and ground char (moisture content less than 0.5 wt%) were prepared by mixing the bio-oil with the wood, char and ground char, respectively. No additional additives were used. Physical and chemical properties of the wood and chars were measured by the following methods: the ash content by ASTM D1102 – 84, the higher heating value (HHV) by a bomb calorimeter (IKA C-200), the elemental contents by flash combustion (Thermo Finnigan, Flash EA1112) for CHN, by an inductively coupled plasma–optical emission spectroscopy, (ICP-OES) axial for Cl and S, and by ICP-OES radial for Al, Fe, P, Si, Ca, Mg, K, and Na, and the oxygen content was calculated by difference. The size distribution was determined by sieve analysis using a sieve with sizes of 45–1000 μm. Pore size distribution was measured by mercury porosimeter (mercury porosimeterpascal 140 and 440, Porotec). The macropores were measured by the pascal 140 equipment (at pressure up 400 kPa), and mesopores and micropores were measured by the pascal 440 equipment (at pressure up to 200 MPa). Bulk density of the wood and chars were calculated by measuring the mass of solid in a certain volume. The wood, char and ground char samples were continuously poured and tapped on a 250 ml graduated cylinder until obtaining a constant volume. The samples were repeated 3 times to determine a standard deviation. The grinding of char is believed to cause no changes to the physical and chemical properties of the char (except the particle size distribution and bulk density). Thus the properties of the ground char were assumed to be similar to the char.

Physical and chemical properties of the bio-oil were measured by the following methods: water content by a Karl Fischer titration (Metrohm-701 KT titrino), the ash content by ASTM D1102 – 84, HHV by a bomb calorimeter (IKA C-200), density by density meter (Anton paar – DMA 4100), the element contents by flash combustion (Thermo Finnigan, Flash EA1112) for CHN, by ICP-OES axial for Cl and S, and by ICP-OES radial for Al, Fe, P, Si, Ca, Mg, K, and Na, and the oxygen content was calculated by difference.

The values of the slurry samples with respect to HHV and elemental contents were calculated based on the chars/wood and bio-oil data with relevant proportions. Bulk density of the slurry samples was calculated by measuring a mass of slurry in a certain volume. The slurry samples prepared with given solid loadings were poured into a 50 ml volumetric flask and kept immobile for an hour for sufficient soaking of bio-oil into the solid particles. The rheological properties of slurries were performed on a rheometer (TA Instruments, Advanced rheometer AR 2000 with a 6 cm parallel plate geometry) at shear rates of 5–300 s^{-1} and temperatures of 25, 40 and 60 °C.

2.2. Experimental setup for pumping test

Pumping tests were conducted using some of the bioslurry samples. A peristaltic pump (Watson-Marlow 520S IP31) with an internal tube diameter of 9.6 mm was used. A schematic diagram of the pump test is presented in Fig. 1. The bioslurry was prepared in a slurry bottle using a given char loading. The slurries were stirred and heated to a given temperature to obtain a homogenous temperature and to keep the char particles well dispersed. A 500 ml tank was pressurized by nitrogen gas before starting the pump. Due to a limitation of the maximum pump pressure, the pressure of the tank was set at 0–6 bar. A relief valve was installed on the top of the pressurized tank to keep a constant pressure during pumping. The slurry bottle, tube and pressurized tank were heated to the investigated temperatures (25–60 °C) by electric trace heating. The bioslurry samples were pumped into the tank by the peristaltic pump within 20–25 min. A pump speed of 5 rpm was used in the tests of slurry samples with various char and ground char loadings, pressures and temperatures.

3. Results and discussion

3.1. Characterization of wood, char and ground char

Entrained flow pressurized gasification reactors are often designed for a relatively short fuel residence time in the reactor. For the bioslurry sample composed of bio-oil and solid (biomass or chars), the bio-oil easily formed tiny droplets that can obtain a high conversion when injected into the gasifier, while the solid particles are required to be of small size to obtain a good conversion [4,5]. Utilization of coal with a particle size of 50–100 μm is required for most gasifiers [11] to obtain a good conversion. However, biomass has been found to be more reactive than coal [14–16]. Fig. 2 and Table 1 show the particle size distributions of the wood, char and ground char samples. The wood had a d_{50} of 300 μm and d_{80} of 392 μm which is believed to be a suitable size for entrained flow gasification [15,16] and probably also for pressurized gasification with a low pressure fluidized-bed gasifiers. The size reduction of wood to less than 400 μm requires a significant power consumption for milling when compared to that of pyrolysis process [4]. Thus the use of wood particles with a size of less than 400 μm is probably not feasible [4]. The wood sample with d_{80} of 392 μm (d_{50} of 300 μm) (see Table 1) was used for the experiment. The char and ground char have a d_{80} of 276 μm (d_{50} of 90 μm) and d_{80} of 118 μm (d_{50} of 45 μm), respectively. The size of the char and the ground char are believed to be a suitable feedstock for entrained flow pressurized gasifiers using pressures up to 20–50 bar.

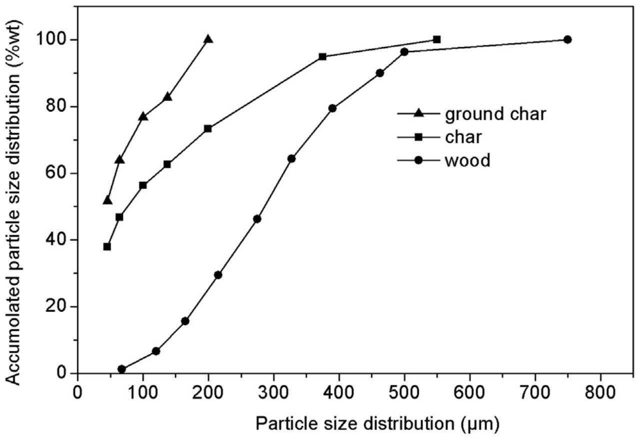

Fig. 2 – Wood and char particle size distributions.

The pore size distributions of wood and char samples are presented in Fig. 3, both samples have a majority of macropores (pore size larger than 0.5 μm) and a minor amount of mesopores (pore size of 0.08–0.5 μm) and micropores (1.8–80 nm). The wood pores mainly appear in two ranges of

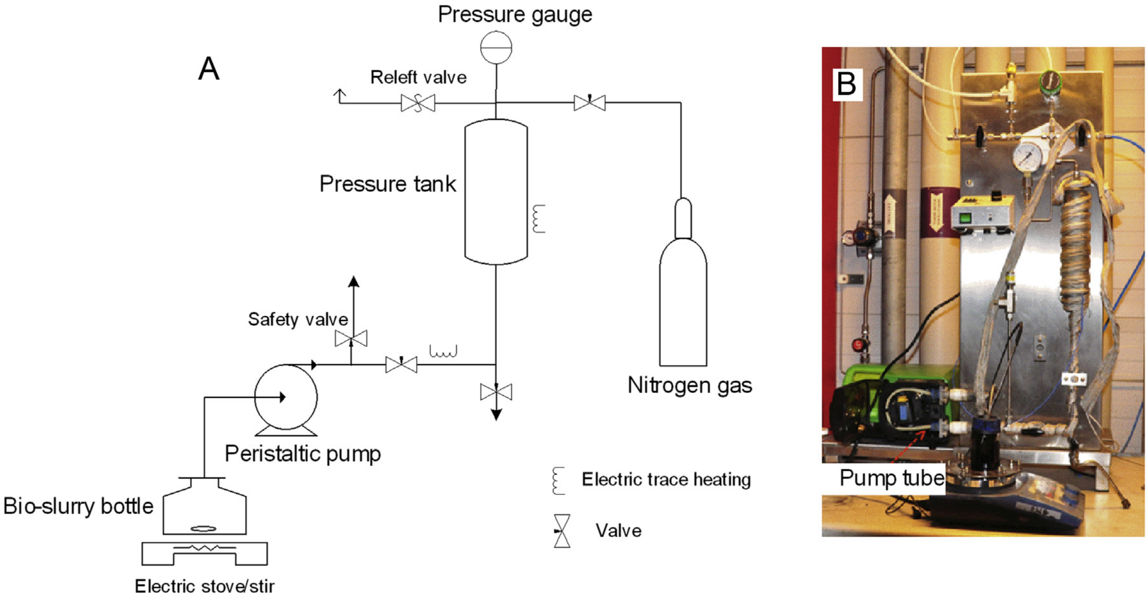

Fig. 1 – Pressurized pump setup (A: PI-diagram, B: picture).

Table 1 – The morphological properties of the wood, char and ground char samples.

	Wood	Char	Ground char
d_{50} (μm)	300	90	45
d_{80} (μm)	392	276	118
Bulk density (kg/m^3)	267 ± 60	362 ± 38	342 ± 13

0.1–20 and 20–100 μm while the char mainly had pore sizes in a range of 3–50 μm. The difference was probably influenced to bio-oil absorption of the char samples and the wood sample.

3.2. *Phase transition and rheological properties of the slurries*

The bioslurry samples are stirred to obtain a homogeneous phase for the phase transition studies. Table 2 shows the phase transitions of the bioslurry samples with solid loadings in a range of 0–25 wt% that were evaluated based on visual observations. The slurry samples with char and ground char transformed from liquid to a paste phase (liquid-paste form) at the solid loadings of 15–20 wt% and become totally solid phase when the solid concentration was 25 wt% (see Fig. 4). While the wood slurry samples have the phase transition at lower solid loadings (10–15 wt% for the liquid to paste change and 15–20 wt% for the paste to solid change). The solid forms are difficult or even impossible to pump into a pressurized reactor. Thus the solid loading of 20 wt% for the char and ground char slurries and 15 wt% for the wood slurry samples were used for the rheological and pump properties investigations.

As can be seen in Fig. 5, the apparent viscosity of the slurry samples with solid loadings higher than 10 wt% for char and ground char, and 5 wt% for the wood were observed to have a non-Newtonian pseudo-plastic behavior (with a good pumpability) in the investigated shear rate range at temperatures of 25, 40 and 60 °C. The investigation of fluid behavior as function of shear rate is important with respect to pumping properties. The phase transition from liquid to paste form takes place rapidly by adding solid from 15 to 20 wt% for the char and ground char slurry samples and from 10 to 15 wt% for the wood slurry sample. The paste phase considerably enhanced the apparent viscosity at all investigated temperatures of 25, 40 and 60 °C.

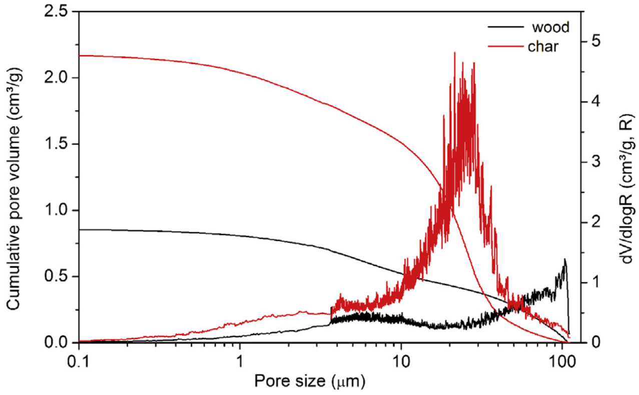

Fig. 3 – Pore size distribution of the wood and char.

Fig. 6 presents the effect of the temperature and solid loading of slurries on apparent viscosity at the shear rate of 100 s^{-1}. At the phase transition region (from liquid to paste form), the slurry viscosities were observed to increase 5–7 times of that of the char and ground char slurries and 10–17 times for the wood slurry. The slurry viscosities of the transition region (the slurry solid just before phase transition to a paste) at temperatures of 25, 40 and 60 °C are 2.4, 0.8 and 0.7 Pa.s for 20 wt% char loading, 3.9, 1.5 and 1.0 Pa.s for 20 wt% ground char loading, and 5.3, 2.2 and 1.7 Pa.s for 15 wt% wood loading, respectively. Increasing slurry viscosity with a decreasing temperature from 60 to 25 °C or increasing solid loading is also seen at lower solid loading levels. The rheological properties with respect to effect of char loading, temperature and apparent viscosity are consistent to those of bioslurry samples from bio-oil and pyrolysis char found by Abdullah et al. [11].

The high degree of oil absorption to the wood compared to the chars caused a rapid increase of the slurry viscosity and made the phase transition occur at a lower solid loading (15 wt % for the paste phase transition and 20 wt% for the solid phase transition). Wood was found to have more hydrophilic properties [17] and the pyrolysis char to be more hydrophobic [18], Bio-oil that is miscible to ethanol and immiscible in petroleum oil has hydrophilic properties, which probably caused the bio-oil absorption in the char to be more difficult than in the wood. Furthermore, the char has a higher amount of small pores (less than 31 μm) but a smaller amount of large pores (more than 31 μm) compared to the wood (see Fig. 3). Thus those could explain the different phase transition of the wood slurry compared to the char slurry samples. The ground char slurry has a higher viscosity than the char slurry at the same solid concentration. The phenomenon was also found by He et al [17] who investigated rheological properties of biomass-water slurries. In order to operate a pump to inject slurry into a pressurized gasifier, the slurry viscosity is expected not to be higher than a few Pa.s [1]. Thus the bioslurry samples with a solid loading of 20 wt% for the char and ground char and 15 wt % for the wood can probably be pumped into a pressurized gasifier at a temperature of 60 °C.

3.3. *The elemental content of the bioslurries*

Pressurized gasifiers are often operated at temperatures of 1200–1800 °C, thus a refractory wall (SiC material) is used to protect the reactor wall against the harsh conditions such as high temperature, corrosion and erosion in the gasifier. The refractory wall is designed to operate with a thick slag layer generated by the gasifing process and the slag layer itself reduces the heat loss from the reactor and protect the walls against high temperature. The viscosity of the slag layer is often required to be about 8–15 Pa.s and not more than 25 Pa.s to maintain a continuous movement of the slag layer into the quench zone [5,19]. Due to the high gasifier temperature, the slag is found to be rich in Ca and Si [19] while other ash forming elements are released in the gas phase. In this study the total CaO plus SiO_2 were 2.7 and 0.33 wt% in the bioslurry (see Fig. 7) at 20 wt% char loading and 15 wt% wood loading, respectively. Thus, in the case of the wood slurry, the amount of slag forming ash provided to a gasifier seems not to be

Table 2 – Visible observation of phase transitions.

Solid load (wt %)	Char loading			Ground char loading			Wood loading		
	Liquid	Paste	Solid	Liquid	Paste	Solid	Liquid	Paste	Solid
0[a]	X			X			X		
5	X			X			X		
7.5	X			X			X		
10	X			X			X		
12.5	X			X			X		
15	X			X				X	
20		X[b]			X[b]				X[b]
25			X[b]			X[b]			X[b]

[a] Bio-oil.

[b] Investigated at 25, 40 and 60 °C.

adequate to protect the gasifier walls to obtain a long service life when gasifying the bioslurry. In order to obtain a safe operation with a low Ca and Si content feedstock, additives such as silica or clay should probably be added to obtain an adequate slag layer coverage.

The slurry samples produced by mixing of 80–85 wt% of bio-oil (having a water content of 25 wt%) and 15–20 wt% char or wood provided slurries that has carbon content of 43–45 wt %, hydrogen content of 6.8–7.4 wt% and oxygen content of 44–48 wt% (see Fig. 7). Thus the wood, char and ground char slurry samples had relatively similar C, H and O contents in comparison to those of beech wood, and have low N (<0.4 wt %), S (<0.03 wt%) and Cl (<0.1wt%) contents. If all the char and bio-oil products from the wood PCR pyrolysis are used to produce a bioslurry, the slurry will have a char concentration of 17 wt% and it has a relatively similar elemental composition to that of beech wood.

3.4. *Energy density of the slurries*

Table 3 shows the mass energy density, bulk density and volumetric energy density. The grinding of char was considered not to change the mass energy density (HHV), the HHV of the ground char is thus assumed to be the same value as that

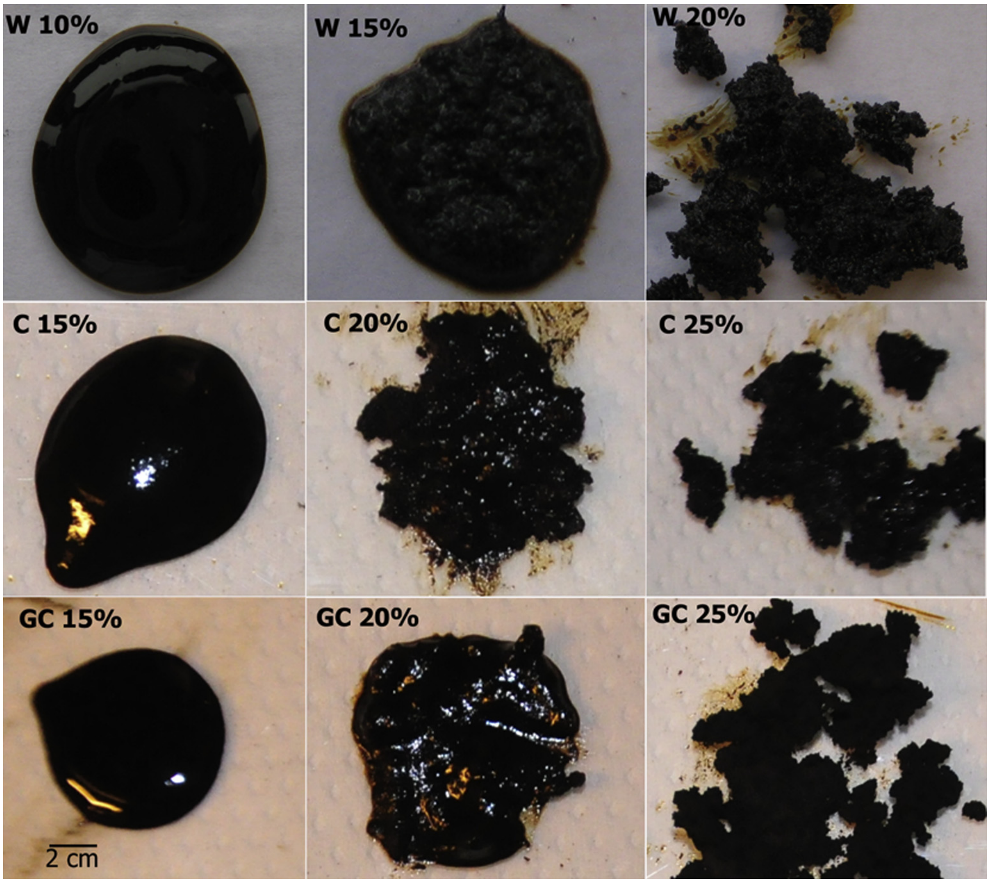

Fig. 4 – Images of the slurries at 25 °C with different solid loading (W: wood slurry, C: char slurry, GC: ground char slurry).

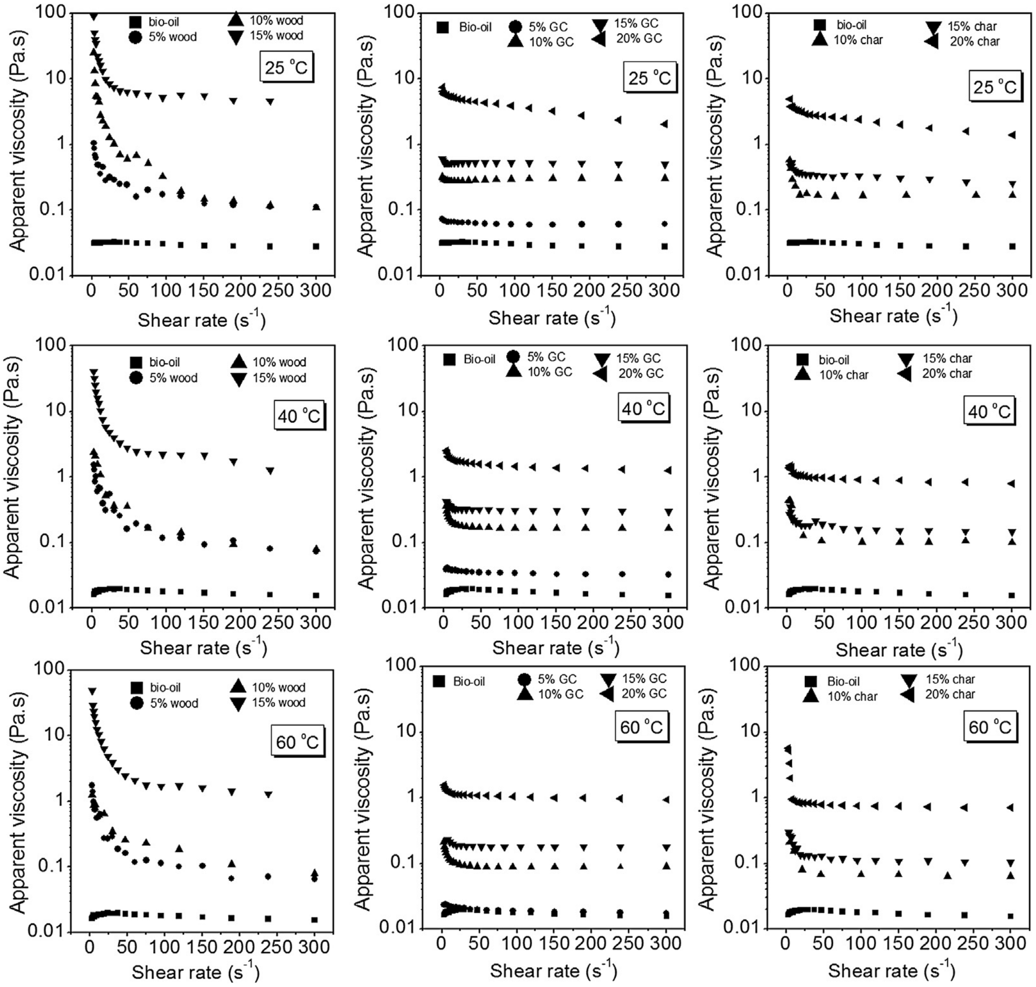

Fig. 5 – The apparent viscosity of bioslurries as a function of share rate at temperatures of 25, 40 and 60 °C (GC: ground char).

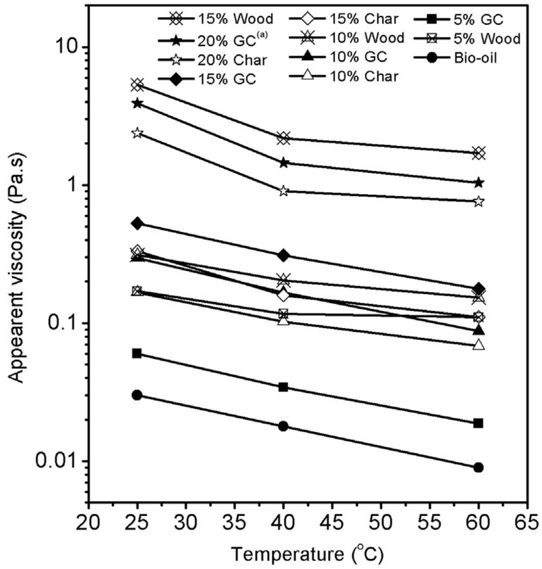

Fig. 6 – Effect of temperature and char loading on bioslurry viscosity at shear rate of 100 s^{-1} (^{a}Ground char).

of the char. The wood and chars slurry's HHVs vary in a limited range of 18.2–18.5 MJ/kg, while their bulk densities increased from 1125 to 1254 kg/m^3 when the solid concentrations increases from 0 to 20 wt%. The densities of the slurries increased by about 7–14% when compared to that of the bio-oil. The volumetric energy densities of the slurries with chars loading of 15–20 wt% are 21–23 GJ/m^3. For a better illustration of the volumetric energy density of the slurries, the energy densification factors (EDF), were calculated as the ratios of the volumetric energy density of the slurries or chars relative to the wood and are presented in Fig. 8. Due to the low wood bulk density of 267 kg/m^3, the wood has a low volumetric energy density of 4.6 GJ/m^3. Consequently, the EDF of the char and wood slurries obtained a 4.5–5 fold increase. The results of the volumetric energy density and EDF of the char slurry are consistent to those reported by Abdullah et al. [11] who studied a bioslurry made from mallee pyrolysis. Biomass fast pyrolysis is, thus, a promising method to produce a high energy volume density fuel. Bioslurries have even a higher volumetric energy density than solid fuel provided by biomass torrefaction in combination with pelletizing (TOP) which produces fuels having volumetric energy densities of 11–19 GJ/m^3 [20].

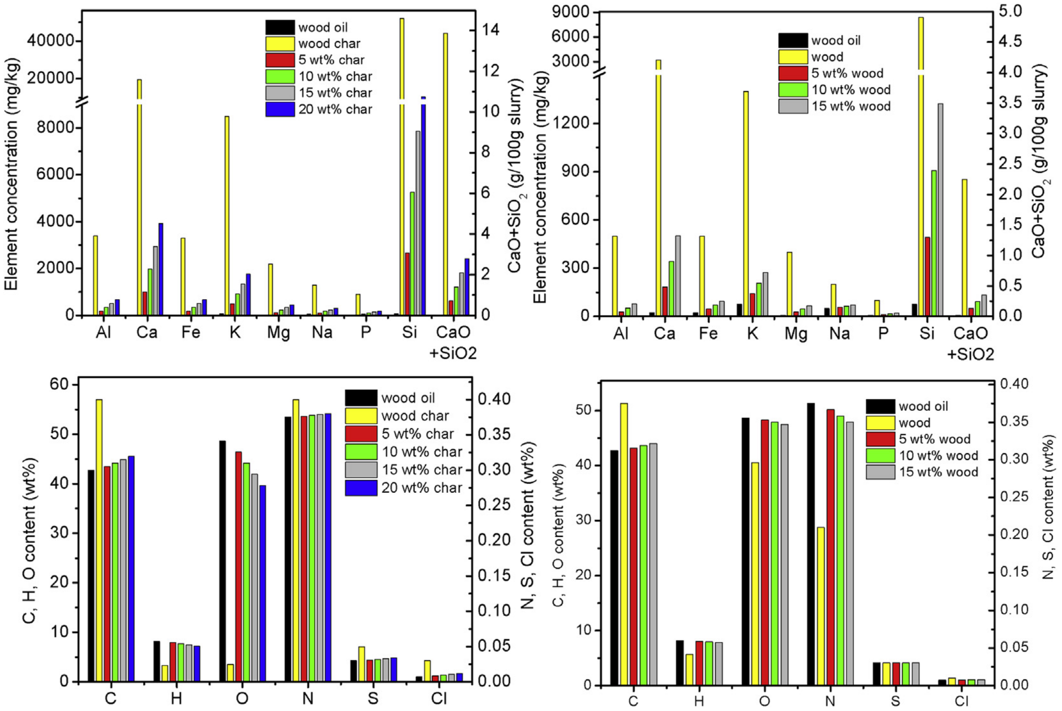

Fig. 7 – The elemental compositions of bio-oil, char, wood and bioslurry.

3.5. *Pumping investigation of char and ground char slurries*

Pumping of slurry is influenced by type and size of the applied pump. However, in this work, the slurries with char and ground char loadings from 0 to 20 wt% which corresponds to viscosity from 30 mPas to 6 Pas (at temperatures of 25–60 °C, see Fig. 6) were investigated using the peristaltic pump test rig at a speed of 5 rpm within pressure of 0–6 bar and temperatures of 25–60 °C. It may be a challenge to pump the slurries due to the high viscosity and large size of the solid particles. The results are presented in Fig. 9. The bio-oil pumping is observed not to be influenced much by the applied temperatures (25–60 °C) and pressure (0–6 bar), and flow rates of 10–11 ml/min are obtained. The pump tests of the slurry samples with 10, 15 and 20 wt% of ground char at 25 °C, and 15 wt% of ground char loading at 40 °C were conducted successfully. The results showed that the flow rates of the slurry samples were not influenced by the char loadings from 0 to 20 wt%, the applied temperatures and pressure treatments as they demonstrated similar values as that of the bio-oil.

A pump test of the slurry sample with 10 wt% char loading at 25 °C was successful, however for a test of the slurry with a char loading of 15 wt% at temperatures of 25 and 40 °C, plugging appeared within 7 min. The plug was identified in a pump tube (having an internal tube diameter of 9.6 mm) (see Fig. 1). The viscosity of the slurry with 15 wt% char loading at 40 °C (0.16 Pa.s) is relatively similar to that of the slurry with 10 wt% ground char loading at 25 °C (0.17 Pa.s) and much lower than the slurry with a ground char loading of 15 wt% at 25 °C (0.52 Pa.s). Thus the block was probably caused by larger char particles that accumulated on the tube wall. This indicates that the char particle size is more important for the pumping

Table 3 – The mass energy density, volume energy density and bulk density of the wood, char and ground char slurry.

Solid loading (wt%)	HHV (MJ/kg "as received")		Bulk density (kg/m^3)			Volumetric energy density (GJ/m^3)		
	Char/G.Char[b]	Wood	Char	G.Char	Wood	Char	G.Char	Wood
0[a]	18.3	18.3	1125	1125	1125	20.6	20.6	20.6
5	18.4	18.3	1152	1144	1152	21.2	21.0	21.1
10	18.4	18.2	1204	1149	1201	22.2	21.1	21.9
15	18.5	18.2	1244	1160	1254	23.0	21.5	22.8
20	18.6	–	1258	1184	–	23.4	22.0	–
100%	19	17.3	362	342	267	6.9	6.5	4.6

[a] Bio-oil with 25 wt% water content.

[b] Ground char.

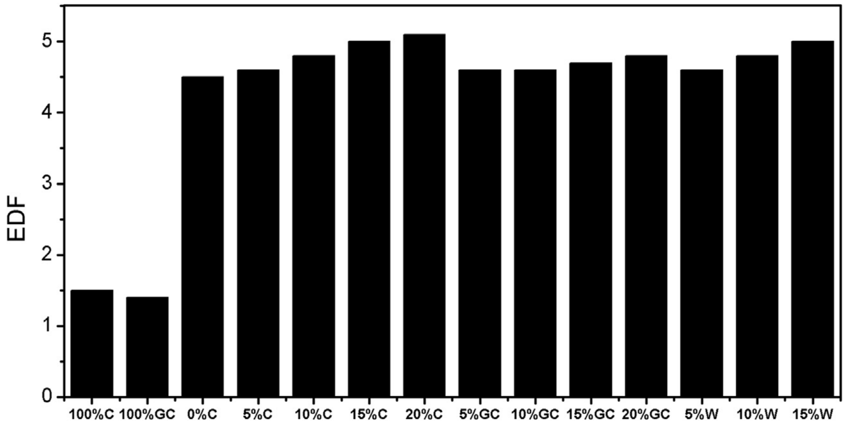

Fig. 8 – Energy densification factors of the bioslurries with various chars and wood loadings (C: char, GC: ground char, W: wood).

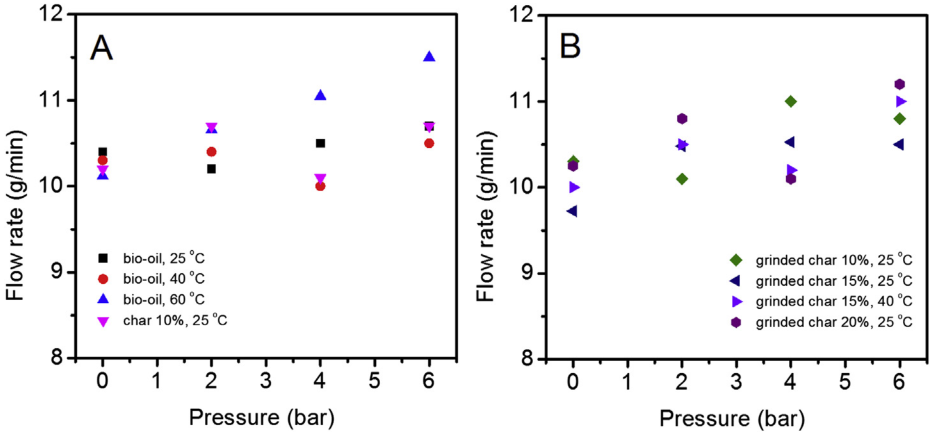

Fig. 9 – The effect of pressure of the pumping on slurry flow rate with bio-oil and char loading (A) and ground char loading (B).

properties than the slurry viscosity with char concentrations of 0–20 wt % and temperatures of 25–60 °C. Using a small particle size of less than 118 μm (d_{80}) is favorable with respect to pumping and this is probably also a suitable size for atomizer nozzles of gasifiers.

4. Conclusions

The properties of bioslurry made of pyrolysis oil mixed with wood, char and ground char were investigated in this study. The slurries with wood, char and ground char with 5–20 wt% solid loadings obtained volumetric energy densities of 21–23 GJ/m^3, consequently considerable volumetric energy densifications (4.5–5 times) were obtained when compared to beech wood. This significantly reduces transportation costs when bioslurry is transported from a biomass slurry production site to a central bioenergy plant as compared to transportation of biomass. The wood, char and ground char slurry samples had similar C, H and O contents to that of the beech wood and had low N (<0.4 wt%), S (<0.03 wt%) and Cl (<0.1wt %) contents. The apparent viscosity of the slurry samples was found to be considerably impacted by solid loading level (0–20 wt%) and temperature (25–60 °C). A slurry sample with 20 wt% ground char loading (having a d_{80} of 118 μm) was pumped successful into a pressurized chamber (0–6 bar) while plugging appeared when using a slurry sample with 15 wt% char loading (having a d_{80} of 276 μm). Thus, the ground char slurry might be considered as a promising feedstock for feeding by pumping into a pressurized gasifier.

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